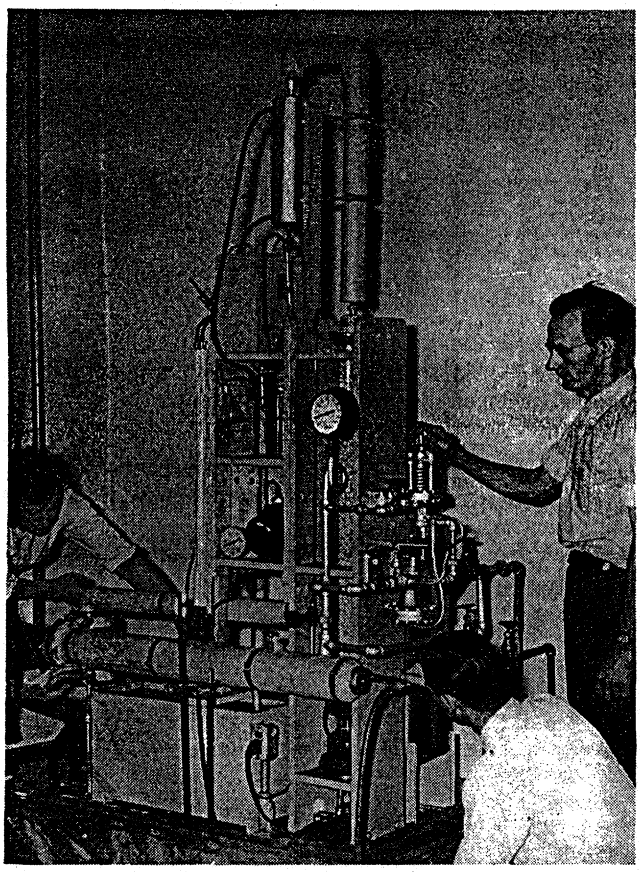
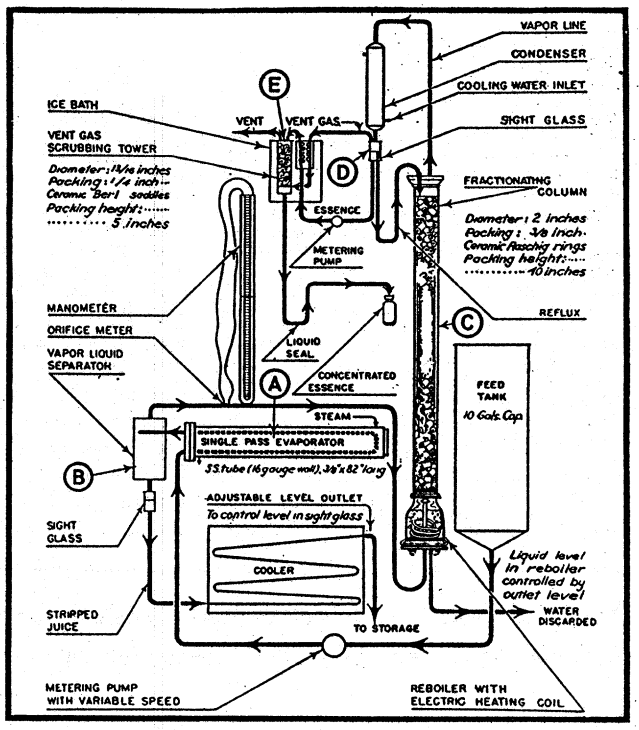


THIS volatile flavor recovery unit, designed for apple juice, is now producing satisfactory essences from other fruits.



HOW flavor recovery unit works: Juice from 10-gal. feed tank is pumped through steam-heated evaporator (A), temperature of which is controlled to produce desired fraction of vapor, then goes to vapor-liquid separator (B). From here, vapor passes to fractionation column (C), where it is concentrated, then passes through water-cooled condenser. Gas, not condensed here, is chilled in an ice bath and stripped by concentrate in scrubber (E).

New Progress In Fruit Flavor Recovery

Many volatile fresh fruit flavors—lost in previous processes—now are preserved by using unit that solved apple problem

EDWARD L. GRIFFIN, Jr., LYLE L. DAVIS, NELSON H. EISENHARDT and MARGARET E. HELLER

Associated with Eastern Regional Research Laboratory, Philadelphia 18, with exception of Author Davis, who is with Virginia Agricultural Experiment Station, Blacksburg, Va.

Today, new capabilities are being demonstrated by a specially developed fruit-essence recovery process for supplying concentrated volatile flavors suitable for use in fruit jellies and candies.

Using a portable flavor recovery unit, this process had already performed notably in capturing the volatile, fragrant constituents of apple juice in concentrated form[1]. Then recently it was applied to other fruit juices—grape, strawberry, blackberry, youngberry, huckleberry, peach and rhubarb. And evaluation of these new essences invites exploration of possibilities for commercial employment of the method.

However, efficiency of the process, also the evaporation required, vary with the juice. With the equipment used, evaporations of 15 to 20 percent were, in general, found to give the best results.

Profitable operation of the process will require use of the stripped juices and possibly the pressed fruit—for value of the essence cannot be expected to carry the total cost of the raw fruit unless it is prepared from waste products. Stripped juices of apples and grapes can be concentrated under vacuum to produce concentrates, which are already accepted as commercial products.

The work reported here was done at the Virginia Agricultural Experiment Station, Blacksburg, Va., in cooperation with the Eastern Regional Research Laboratory. Used was a small—10 gal. per hr.—portable essence recovery unit, designed and built by the ERRL[2].

Juices were prepared from frozen fruits collected during 1946 and 1947. The frozen materials were removed from storage and thawed overnight in their containers. To avoid altering the flavor, the fruits were not heated before pressing, but the freezing and thawing no doubt disrupted the cells to some extent.

The thawed material was pressed through a heavy grade of muslin in a hand-operated cider press and screened

[1] Milleville, H. P. and Eskew, R. K. "Recovery and Utilization of Natural Apple Flavors," USDA, Bureau of Agr. and Ind. Chem. AIC-63 (Eastern Regional Research Laboratory) 1944, with Supplement 1; April, 1945.

[2] Milleville, H. P. and Eskew, R. K. "Recovery of Volatile Apple Flavor in Essence Form," *Western Canner and Packer*, Vol. 36, 51-4, October, 1946.

to remove coarse particles of the fruit.

Raw materials used in the experiments were:

Grapes: Moore's Early, Niagara, Delaware and Worden. Grown at the Virginia Agr. Expt. Sta. in 1947, washed and stemmed. Skins and pulp were separated, frozen and stored at 0 F.

Strawberries: Blakemore, with caps. Grown at Virginia Truck Expt. Sta., Norfolk, in 1947; rejected by freezer plant because of excessive rot, probably *Botrytis*. They were frozen in 50-lb. tins and stored at 0 F.

Strawberries: Blakemore, with caps removed. Discards from the Chickamauga Cooperative Frozen Food Plant, Dayton, Tenn. Berries were washed, capped by machine, and sorted on an inspection belt. Discards were frozen in 50-lb. tins, and stored at 0 F.

Peaches: Elberta. From the 1947 pack delivered to Crozet Cold Storage Corp., Crozet, Va. They were lye peeled, washed, neutralized, inspected, and sliced. Sugar with ascorbic acid was added to make a commercial 4-to-1 pack; they were frozen, and stored at 0 F.

Blackberries: Wild. Harvested in Grainger County, Tenn., in 1946, and delivered to the Winter Gardens Frozen Food Corp., Knoxville. They were frozen without washing in 50-lb. tins, and stored at 0 F.

Youngberries: Grown near Dayton, Tenn., in 1946, and delivered to TVA Experimental Frozen Food Barge near Spring City. They were washed, sorted, frozen individually in glycerine, packed in 2-lb. cellophane bags overwrapped with single waxed boxes, and stored at 0 F.

Huckleberries: Wild. Harvested near Jamestown, Tenn., in 1946, and delivered to the Winter Gardens Frozen Food Corp. They were frozen without washing or stem removal in 50-lb. tins, and stored at 0 F.

Rhubarb: Victoria. Grown at Blacksburg in 1947. It was washed, cut into ¾ to 1½-in pieces, frozen in 50-lb. tins, and stored at 0 F.

Grape skins and pulp were pressed separately, and their juices combined for processing, except those from Moore's Early, which were processed separately. In each case, juice from the skins had a good flavor, characteristic of the grape used. That from the pulp was bitter and lacked a good grape character.

Peaches were pressed again, after admixture of the pulp with water equal to 98 percent by weight of the expressed juice. Table I shows percent extraction, pH value, and degrees Brix of the juices.

Apple Essence Equipment Employed

The unit used in the experiments was designed to produce 150-fold apple essence from 10 gal. of apple juice per hour (see flow diagram). In the standard process for apple juice, 10 percent of the juice is evaporated in less than 20 sec. in evaporator (A). It is separated from the liquid at (B) and

Detailed Studies Give Well-Rounded Checkup On End-Products

TABLE I—RECOVERY of Essences From Fruit Juices

Fruit	Variety	Lot	Juice Brix Deg.	Juice pH	Extraction Percent	Evaporation Percent	Theoretical Conc. of Essence Fold	Aroma in Bottoms [1]
Grape	Worden		11.2	3.23	71.2	18.2	108	Slight
Grape	Niagara		13.4	3.35	73.0	18.2	112	Slight
Grape	Delaware		18.0	3.40	76.2	18.2	112	Slight
Grape	Moore's Early	Skins	12.2	3.45	88.1 [2]	18.2	101	Slight
Grape	Moore's Early	Pulp	12.0	3.15	42.4 [2]	18.2	105	Slight
Strawberry	Blakemore	No caps	9	3.73	72.2	18.2	108	Slight
Strawberry	Blakemore	With caps	5.8	3.70	83.0	20.0	89	Slight
Peaches	Elberta	1st pressing	24	4.05	44.6	18.2	100	Very slight
Peaches	Elberta	2nd pressing [3]	13.2	4.10	42.3	18.2	99	None
Huckleberry	Wild		18	3.60	67.2	18.2	96	None
Blackberry	Wild		8.9	3.62	68.9	20.0	90	None
Youngberry			12.0	3.30	65.6	18.2	104	None
Rhubarb	Victoria		4.2	3.38	89.5	18.2	112	None

[1] Water discarded from base of fractionation column.
[2] Over-all extraction of Moore's Early Grapes was 67 percent.
[3] Water added to first pressing equal to 98 percent of juice from first pressing.

TABLE II—STRIPPED JUICES Lacked Fresh Flavor, But Reconstituted[1] Ones Were Similar to Fresh Juices

Fruit	Variety	Stripped Juice [2]	Reconstituted Juice [3] as Compared With Fresh Juice
Grape	Worden	Very little aroma	Very little aroma
Grape	Niagara	Very little aroma	Sl. better flavor
Grape	Delaware	Sl.[4] heavy aroma	Blander, sl. cooked aroma
Grape	Moore's Early (skins)	Sl. heavy aroma	Sl. heavy aroma
Grape	Moore's Early (pulp)	Sl. heavy aroma	Sl. sweeter
Strawberry	Blakemore	Preservelike aroma	Sl. weaker aroma
Peaches	Elberta	Sl. preserve aroma	Sl. " canned " peach flavor
Huckleberry	Wild	Sl. preserve flavor	Sl. blander
Blackberry	Wild	Sl. preserve flavor	Sl. preserve flavor
Youngberry		Little aroma	Sl. weaker aroma
Rhubarb	Victoria	Cooked aroma	Sl. less fresh

[1] Mixture of stripped juice, essence and water in same proportion as in fresh juice.
[2] All stripped juices lacked fresh flavor.
[3] All reconstituted juices were similar to the fresh juices. Slight differences are listed here.
[4] Slightly.

TABLE III — FLAVOR CONCENTRATES Were Evaluated to Distinguish Between Fractions

Fruit	Quality: Top Note	Quality: Undertone	Strength
Strawberry, Blakemore	Excellent	Fair	Good
Peaches, Elberta	Good	Good	Good
Huckleberry, wild	Good	Good	Good
Rhubarb, Victoria	Good	Good	Good
Blackberry, wild	Fair	Poor	Fair
Youngberry	Good	Fair	Fair
Grape, Worden	Excellent	Fair	Good
Grape, Niagara	Excellent	Fair	Good
Grape, Delaware	Fair	Poor	Fair
Grape, Moore's Early	Excellent	Fair	Good

TABLE IV—JELLIES, Prepared With Stripped Juices and Various Quantities of Essence[1], Were Scored Organoleptically

Concentration of Essence, Fold [2]	0		0.3		0.6		0.9	
Storage, Time, Months [3]	3	6	3	6	3	6	3	6
Grape, Worden	7.3	8.0	8.8	8.7	9.6	8.5	8.1	8.6
Grape, Niagara	7.8	7.5	8.6	8.9	9.1	9.0	8.3	8.4
Grape, Delaware	8.1	8.4	8.6	9.0	9.1	8.5	8.0	8.0
Grape, Moore's Early	8.0	8.4	9.2	8.6	9.0	8.5	7.7	8.3
Strawberry	8.1	8.0	8.8	9.0	8.5	8.8	8.5	8.1
Huckleberry	7.8	8.1	8.5	9.0	9.0	8.6	8.6	8.1
Blackberry	8.1	8.3	8.0	9.0	10.0	8.8	7.6	7.7
Youngberry	7.8	7.9	8.6	8.2	9.5	9.1	8.0	8.6
Rhubarb	8.3	8.0	8.3	8.6	9.1	9.0	8.1	8.1
Rhubarb — blackberry [4]	8.1	8.0	9.5	8.7	8.8	9.0	7.5	8.1

[1] Rated on a scale of 7 to 10 by a panel at Blacksburg, Va.
[2] Fold of essence in jelly is its concentration relative to its concentration in original juice by weight. An approximate figure only, this varies slightly from jelly to jelly.
[3] Stored at room temperature.
[4] Prepared from stripped rhubarb juice and blackberry essence.

TABLE V—FLAVORS of Strawberry and Youngberry Were Preferred Among Various Candies and Jellies Prepared With Fruit Essence[1]

Essence	Candy Score [2]	Jelly Score [3]
Strawberry	7.6	8.3
Youngberry	7.4	8.3
Grape, Moore's Early	6.9	7.8
Grape, Worden	6.8	7.7
Grape, Niagara	6.6	7.7
Huckleberry	6.6	6.7
Peach	6.2	7.5
Blackberry	6.0	7.0
Grape, Delaware	5.7	—
Rhubarb	5.2	6.1

[1] Scored on a scale of 1-to-10 by a panel of 25 at Philadelphia.
[2] Pectin gum candy, 2-fold essence added.
[3] Jelly containing 65 percent sugar, 1-fold essence added.

concentrated in fractionation column (C).

The fraction evaporated is measured by a manometer, across an orifice in the vapor line. Rate of evaporaton is controlled by adjusting the steam pressure on the evaporator. The noncondensable vent gas is chilled in an ice bath and stripped by passing it counter-currently with the chilled product in scrubber (E). Volumetric concentration of the product is controlled by the rate it is pumped from chamber (D) to the scrubber. Thus, if one part of the product is pumped off for every 100 parts of juice run to the evaporator, the resultant essence contains all the aroma recovered from 100 parts of fresh juice and has a 100-fold volumetric concentration.

The fractionation column has a maximum vapor capacity of about 1.5 gal. of water evaporated per hour. For this reason, when 20 or 30 percent fractions are evaporated, the feed rate is reduced from 10 gal. to 5 or 3½ gal. per hr., respectively, so that the volume of vapor to the fractionation column remains constant (1 gal. per hr. evaporated in each case). Since the unit was not designed for these conditions, it will not necessarily give optimum performance under them. In order to get the minimum heat damage, the evaporation should be designed for the conditions used.

Experimental Procedure

The general procedure was first to determine approximately the evaporation required to recover the most desirable essence, and then, using this evaporation, to produce essences of about 100-fold volumetric concentration. Approximately 10, 20 and 30 percent fractions were evaporated, and condensed without concentration. These fractions were diluted with water to a volume equal to that of the fresh juice from which they were produced.

Diluted fractions and their respective stripped juices were compared by sense of smell with the fresh juices to determine completeness of stripping off of aroma, possible alterations in character, and relative strength and quality. In all cases tested, the stripped juice after 10 percent evaporation had a slightly fresher flavor than the stripped juices after 20 or 30 percent evaporation, which had about the same strength.

There seemed to be a heat-developed aroma in some of the stripped juices after 30 percent evaporation. This might have been caused by the low feed rate used. With rhubarb juice, a slight "cooked" flavor developed at 20 percent evaporation. Based on these results, 18 to 20 percent evaporation was used for all essences produced. This variation was unintentional, resulting from a difference in calibration of the feed pump rate.

Because of the small quantities of huckleberries, peaches, Delaware grapes, and Moore's Early grapes available, it was not possible to determine their optimum evaporation percentages. Table I gives the theoretical concentration of the volatile flavor and a qualitative statement of the aroma of the water discarded from the bottom of the fractionation column. All essences were water white.

Maximum concentration of volatile flavors obtainable was not studied.

Serious fouling of the evaporator tube or foaming in the separator was not encountered with any juice.

Three separate series of organoleptic tests to evaluate the flavor concentrates were made by different groups of people. Different methods of preparation and scoring were used in these tests. Comparison of ratings is valid, therefore, only within the same series. The first of these tests, made at the time of processing, attempted to determine the efficiency of recovery of the flavor from each juice. A second series, carried out later at Blacksburg, compared jellies prepared from stripped juices with various amounts of added essence. The third series, made at Philadelphia, compared for general acceptance standard pectin candies and jellies prepared with the various essences (see Table V).

■ "Particular importance attaches to the accomplishments recounted here—because for the past 25 years the goal of researchers has been capture of the aroma of fresh fruits or their juices without changing the ingredients or their relationship to one another. Yesterday, the goal was reached for apples. And now today other fruits are falling into line."

All essences and stripped juices were evaluated organoleptically for quality and strength of flavor. Each essence was diluted with water to the same concentration as that in the original juice. It was also diluted with stripped juice and sufficient water to reproduce the proportions of the original juice. These samples were then compared with the original juice. Characteristics of the stripped and reconstituted juices are summarized in Table II. The degree to which the aroma of the diluted essence and the flavor of the reconstituted juice approached that of the fresh juice in quality and strength was taken as a measure of the efficiency of the process.

The more fragrant and lighter fractions of the aroma were more volatile and far more readily recovered than the heavier and more pungent undertones. This led to some distortion of the balance between these factors in the flavor concentrates produced. Table III summarizes the evaluation of the flavor concentrates, attempting to distinguish between recovery of the lighter fraction or "top note" and the heavier "undertones" as well as strengths. When these values approximated full recovery of the aroma from the juice, an excellent rating was given, because an essence cannot be expected to be any better than the juice from which it was recovered.

"Top Note" Fractions Easily Recovered

Grape essences contained the top fragrance of the grapes but were lacking in the heavier undertones of aroma. Essences from the Worden, Niagara and Moore's Early grapes were characteristic of the individual varieties, but essence from the Delaware grapes was of poorer quality.

Strawberry essences were slightly weaker than their theoretical concentration and lighter in character than the aroma of the fresh juice. Strawberry essence is apparently composed largely of the "top note" of the fresh strawberry aroma, which is normally lost in processing. Although the strawberries used were discards from commercial processing, the essences were surprisingly good; the essence from the lower-grade rejected berries with caps was only a little poorer than that from the discards without caps.

Peach essence produced from first press juice had a strong fresh peach aroma with an almond-like character typical of the pits. Curiously enough, an essence produced from the juice of the second pressing was very similar in strength as well as in character to that from the first pressing.

Huckleberry essence had a characteristic huckleberry aroma of good strength but of a slightly lighter character than that of the fresh juice.

Blackberry essence, in the concentrated form, was recognizable as blackberry but was lacking in the heavier portion of the aroma. When diluted to juice strength, it was weak and not recognizable as blackberry.

Youngberry essence had a good fragrant aroma, but when diluted with water to its strength in juice the aroma was less pronounced and the mixture had a slightly different character.

Rhubarb essence had a sharp characteristic aroma but was slightly different in character from that of the fresh juice, probably due to development of a cooked flavor.

Jellies were prepared from the stripped juices by adding sugar to 65 percent, bringing to a boil, adding commercial apple pectin, pouring into 250-ml. beakers, cooling to 170 F., adding various quantities of essence

at that temperature, and covering with melted paraffin. A control sample for each fruit was made without essence.

These jellies were set aside on a laboratory shelf at room temperatures, and after 3 and 6 months were tested organoleptically by the same panel of 6 men and 6 women (Table IV). An arbitrary value of 7 was assigned to each jelly that scored the lowest in taste and/or aroma tests. Scores of 8, 9 and 10 were given to the other three samples in order of improving quality.

In general, jellies with 0.3 to 0.6-fold added essence were preferred to those without essence or with 0.9-fold added essence. There appears to be a limit as to how much added essence is desirable. If this should work out in practice, it is obvious that all the essence recovered from a juice will not be required for a jelly, leaving some for other use.

Even after 6 months, the essences could be easily detected in the jellies. Although there are some differences between the ratings at 3 and 6 months, these differences are not consistent and are not sufficient for any significant conclusions. The jelly prepared from stripped rhubarb juice and blackberry essence had much of the flavor of blackberry but retained the rhubarb color. A jelly made from 50 percent rhubarb and 50 percent blackberry stripped juice with blackberry essence was only slightly different from one made from blackberry stripped juice and blackberry essence.

Candy and Jelly Tests

Tests were made at Philadelphia to determine the palatability of the flavors contributed by the essences to pectin gum-type candies and synthetic, 65-percent-sugar jellies prepared without stripped juice. The candies were made from sugar, corn syrup, citric acid, pectin, sodium citrate and coloring matter, and essence added to a 2-fold concentration. The jellies were prepared from sugar, citric acid, pectin, sodium citrate, coloring matter, and essence added to a 1-fold concentration.

These products were submitted to a panel of about 25 people, who scored them on a 1-to-10 scale (10-9, excellent; 8-7, good; 6-5, fair; 4-3, poor; 2-1, objectionable). Table V lists the results in order of preference for the candy. In general, very agreeable flavors characteristic of the fruits from which they were produced were contributed by the essences, and the resulting products were favorably received.

There is a fair degree of agreement between the ratings for the essences in the two products. Youngberry and strawberry flavors were the most popular; only rhubarb jelly and rhubarb, Delaware grape, blackberry, and peach candies rated lower than 6.5. These scorings represent preferences for the individual fruits rather than degree of recovery of volatile aroma from the juices used. It appears likely that more than the optimum quantity of essence may have been used in the jellies produced in Philadelphia. It should be considered, however, that the stripped juices were not used in these jellies. The gum candies normally require a higher proportion of essence than the jellies.

The authors are indebted to G. Macpherson Phillips, Roderick K. Eskew and J. J. Willaman for general guidance in the project reported above.